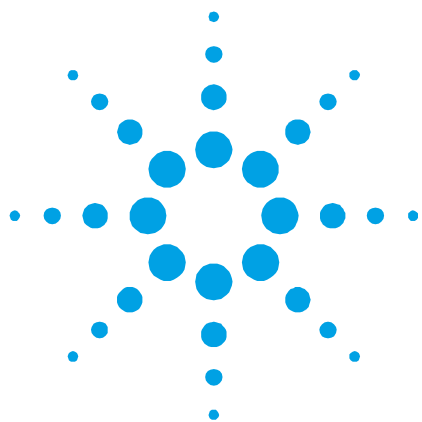




Temperature dependent UV-Vis spectroscopy on polymer thin films; a LINKAM THMS600 hot-stage as sample holder for the Cary 5

Application Note

Author

Edward J. Vlietstra and
Leonardus W. Jenneskens

Debye Institute, Dept of Phys.
Org. Chem., Utrecht
University, Utrecht, The
Netherlands

Introduction

In our group, a Cary 5 UV-Vis-NIR spectro-photometer is routinely used to measure solution and solid state spectra. Here we show that a LINKAM THMS600 hot-stage originally designed for application in optical microscopy can be readily used as a sample compartment in the Cary 5 to obtain UV-Vis absorption spectra of polymer thin films under carefully controlled conditions (temperature and atmosphere).

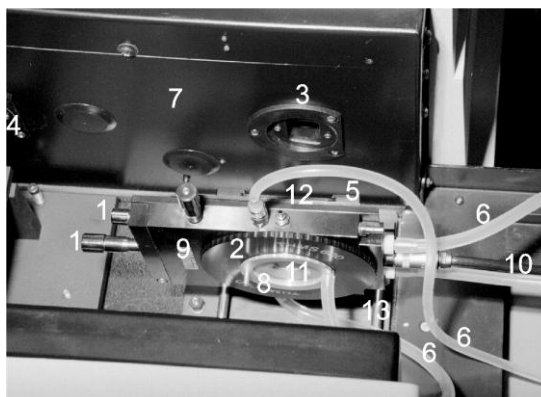


Figure 1. Schematic top view of the hot-stage in the Cary 5 sample compartment

- | | |
|----------------------------------|--------------------------------------|
| 1. N ₂ vents | 8. Quartz window with sample |
| 2. Ag heater block | 9. Hot-stage housing |
| 3. Sample light beam | 10. Electronic- and power-connection |
| 4. Reference light beam | 11. Sapphire window |
| 5. Solid sample slide holder | 12. Adaptor |
| 6. Cooling tubes of heater block | 13. Optical rail |
| 7. Sample compartment | |



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Equipment

The hot-stage is mounted vertically to a solid sample slide holder (5) upon optical rail (13) using a home-built adaptor (12, Figure 1). This X-Y adjustable arrangement allows reproducible positioning of the hot-stage. Although the small sapphire window (diameter 2.5 mm, 11) is optically transparent in the UV-Vis wavelength region (180-800 nm), all standard glass windows have to be replaced by quartz windows (diameter 22 mm and thickness 0.5 mm). A reduced slit height parameter setting is required to focus the sample light beam (3) on the small sapphire window (11). Despite the fact that the beam (3) is partly blocked, photometric noise remains excellent (0.0007 A with and 0.0005 A without the hot-stage).

Discussion

Although the LINKAM THMS600 hot-stage has been designed for use between -200 and 600 °C, it can be conveniently used in the temperature range -100-300 °C using the standard Linkam temperature control unit TMS91. Temperatures down to -100 °C can readily be attained by means of a dry N₂-gas flow cooled with liquid nitrogen. To avoid blocking of the quartz windows by condensation of moisture the double entrance/exit-windows, as well as the internal atmosphere of the hot-stage, have to be purged with dry N₂-gas.

Examples

In Figure 2, the thermochromic behavior of the σ - σ^* transition of a polydihexyl-silylene ($[-Si(C_6H_{13/2})-]_n$) thin film in the temperature region 24.9-42.4 °C is shown. As evidenced by DSC and powder diffraction silicon-backbone conformational changes, i.e. distortion of the all-trans structure, due to hexyl side-chain, melting occurs leading to a hypsochromic shift of the σ - σ^* transition concomitant with increasing temperature¹.

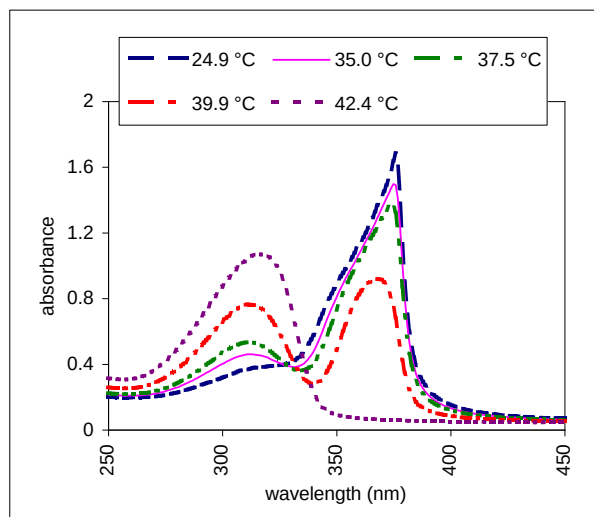


Figure 2. Thermochromic behavior

In Figure 3, the birefringence of a polydihexyl-silylene thin film in the same temperature region is shown. To this end the transmission (%) at 600 nm with the hot-stage positioned between crossed polarizers is monitored (the polymer does not absorb at this wavelength).

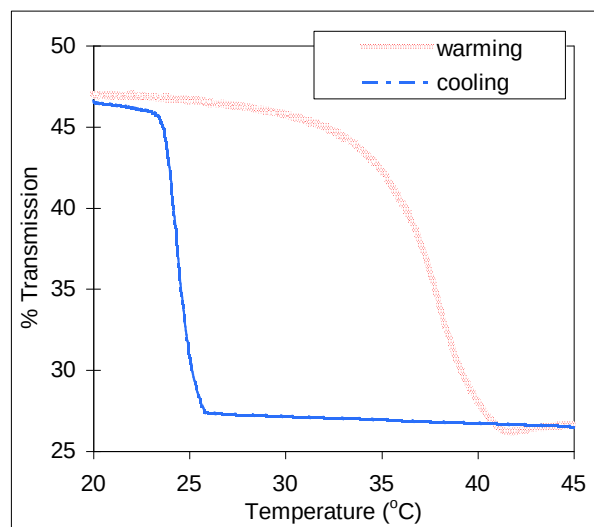


Figure 3. Birefringence

Conclusion

For temperature dependent UV/Vis absorption spectroscopy of polymer thin films the LINKAM THMS600 hot-stage can be readily used as a sample compartment for the Cary 5.

Reference

1. R.D. Miller and J. Michl. Chem. Rev., **(1989)**, 89, 1359.

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